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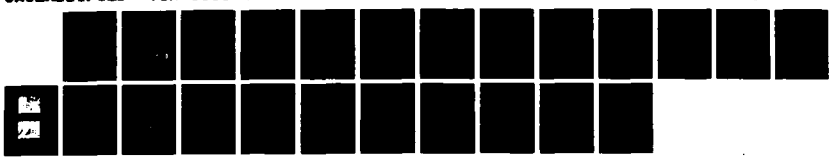

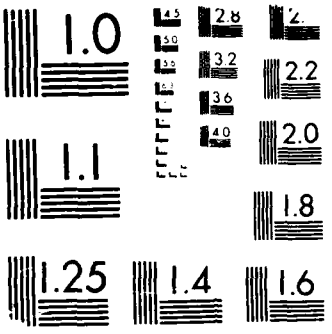

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Analysis of Debris from Accelerated Life Test of the Hughes Five-Year Vuilleumier Cryocooler

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29 May 1987

Prepared for

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AIR FORCE SYSTEMS COMMAND
Los Angeles Air Force Station
P.O. Box 92960, Worldway Postal Center
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Surveillance Technology Development

The information in a Technical Operating Report is developed for a particular program and is therefore not necessarily of broader technical applicability.

ABSTRACT

Unit S3 of the Hughes Vuilleumier (VM) Cryocooler Program has undergone accelerated life testing at twice the service speed expected in a normal unit. Debris samples were obtained from all areas of the cooler; samples of the regenerator balls were collected from the second and third stages only. The debris has been identified by an investigative analysis using the analytical techniques of X-ray diffraction, IMMA, and SEM/EDXS. Most of the particulates contain a combination of Fe, Cr, and Ni or Mo-Nb compounds.

ACKNOWLEDGMENT

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I. INTRODUCTION

Recently, accelerated life testing of unit S3 of the Hughes Aircraft Company's Five-Year Vuilleumier (VM) Cryocooler was completed (26 September 1986). The test consisted of operating the unit for a total of 22,000 hours at twice the speed (300 rpm) of a unit in actual service. As was the case for unit S2 used in the accelerated contamination test (Ref. 1), several stoppages and working fluid exchanges/replenishments occurred during the course of testing and might have affected the overall performance of the cooler. These stoppages have been described in several Hughes reports (Refs. 2 through 5). The S3 unit has now been dismantled for overall inspection and analysis of wear debris generated during the test. The Aerospace Corporation's Materials Sciences Laboratory (MSL) obtained a group of powder samples (see Table 1) for analysis by X-ray diffraction (XRD), scanning electron microscopy with energy dispersive X-ray spectroscopy (SEM/EDXS), and ion microprobe mass analysis (IMMA). Samples of the regenerator balls from the second and third stages were also obtained for examination in the SEM and IMMA.

Table 1. Debris Samples Obtained from Unit S3

Sample No.	Location
3	Top of drive mechanism, 60° from magnet
5	Cylinder #2, circular insert, link-lock cover, right side
7	Between seals between cold cylinder and hot cylinder (45°)
10	Periodic marks on "K" seal
15	Crankcase cover above drive, inside top
18	Under O-ring near cold cylinder link-lock cover
23	"Snowdrift" dèposit, hot cylinder #1
48	First-stage rider, hot side of cold end
49	Second stage, between riders
50	First-stage displacer, near seal at 90°
55	First-stage seals, behind
58	Cross hot rider, displacer #1 at 90°, hot cylinder #1
59	Hot displacer #1, rider at 100°
62	Inside hot cylinder #1 rider
65	Liner #1, near hot rider track
73	Hot cylinder #2, inside
75a	Flake from hot displacer #2, near hot rider at 90°

II. EXPERIMENTAL PROCEDURES

Two methods were used for obtaining powder debris samples: (a) abundant deposits were scraped off surfaces onto pieces of waxed weighing paper, which were then carefully folded and stored in plastic containers, and (b) sparse deposits were removed from surfaces with Scotch Magic Tape and placed face-up in plastic containers. Optical photographs were taken by Hughes personnel of each of the sample areas from which debris was collected, and the location of each sample was documented. The samples and locations are listed in Table 1.

Each of the twelve large granular samples was divided into several fractions for study by different analytical methods. X-ray diffraction was performed first. Samples were finely ground in a mortar, poured into glass capillaries, and placed in a Debye-Scherrer camera. The camera was mounted on a Philips 3100 XRD unit, and chromium K_α radiation was used to irradiate the samples for several hours. The diameters of the diffraction rings on the resultant films were measured and converted to interplanar spacings. The materials producing the patterns were identified by computer matching with the JCPDF (Joint Committee on Powder Diffraction) X-ray diffraction data file. This method gives information about the crystal structures of materials, so that both crystalline compounds and elements present in the unknowns can be identified unequivocally.

Ion microprobe mass analysis (IMMA) was performed on one sample from the hot cylinders in order to complement the results obtained using the other techniques. The debris was mounted on a cylindrical stub using silver paint. The sample was scanned using a primary beam of $^{18}O_2^+$ ions accelerated to 20 kV. A fraction of the sputtered material was ionized, and the positive secondary ions were mass analyzed by the magnetic spectrometer. The intensities of the characteristic peaks were recorded as a function of mass and were identified by comparison with standard tabulated values. This technique allows high sensitivity analysis for identification of trace amounts of material.

All of the powders and tapes were examined using a JSM 840 scanning electron microscope (SEM). The model 9900 EDAX (energy dispersive X-ray analyzer) attachment was used to identify elements present in the various samples. Powder samples were liquid-dispersed on filter paper, dried, and mounted on 1-in.-diameter carbon sample holders with double-stick tape. Tape specimens were cut into small squares and secured to the carbon stubs with Aquadag. All samples were carbon-coated to reduce the effects of charging during exposure to the electron beam. SEM photographs were taken of each sample, using accelerating potentials of 20 and 35 kV. Major elements in each sample were identified by the position of the characteristic K or L X-ray emission energies.

The regenerator balls obtained from the second and third stages were also examined in the SEM and in the IMMA. For the IMMA analysis, the balls were affixed to an aluminum sample mount with silver paint. The operating conditions for the IMMA were the same as those used for the powder debris.

III. RESULTS

The Debye-Scherrer films are not reproduced in this report, but the results are as follows: Sample 7, taken from between seals between the cold and hot cylinders, is ZnO; samples 15, 18, 48, 49, 50, and 55 are $Fe_2F_5 \cdot 7H_2O$; and samples 23, 59, 65, 73, and 75a appear to be amorphous (only a few weak lines or no identifiable lines). Amorphous-appearing XRD patterns occur when crystallite sizes are 100 nm or less. The SEM/EDXS results showed that samples 49, 50, 55, and 62 consist primarily of an Fe-Cr-Ni material, whereas samples 48, 59, and 65 are mostly Mo-Nb compounds with some Fe-Cr-Ni. Debris typical of that seen in all stages is shown in Fig. 1. Rounded metal particulates and pieces of glass fiber can be seen in this photograph. When examined in the IMMA, sample 73 was found to consist of Fe, Ni, Cr, Mo, Nb, Al, and Ti. Samples 3, 5, and 10 were not analyzed by MSL.

Several second-stage regenerator balls are shown in Fig. 2. There appears to be a film on the balls, and there is some evidence of deformation of the surfaces where the balls were in contact. The protrusions evident on the balls appear to be Pb when examined in EDXS. The IMMA charts obtained from the second- and third-stage regenerator balls are shown in Figs. 3 and 4, respectively. Both patterns show Si, F, Pb, Sb, Fe, and Cr. The pattern for the second stage also contains Sn.

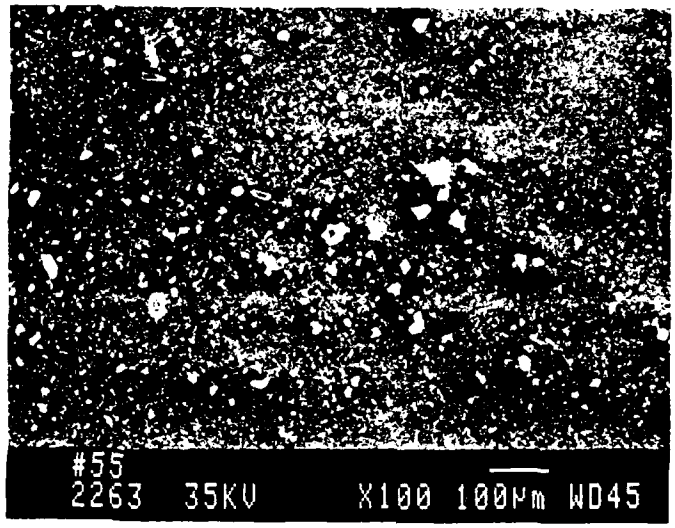


Figure 1. SEM Photograph of Debris Typical of That Observed on All Stages

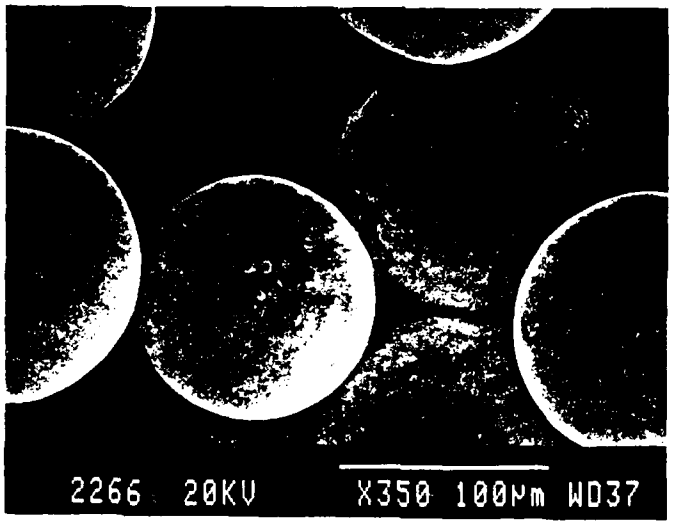


Figure 2. SEM photograph of Second-Stage Regenerator Balls. Blemishes are Pb.

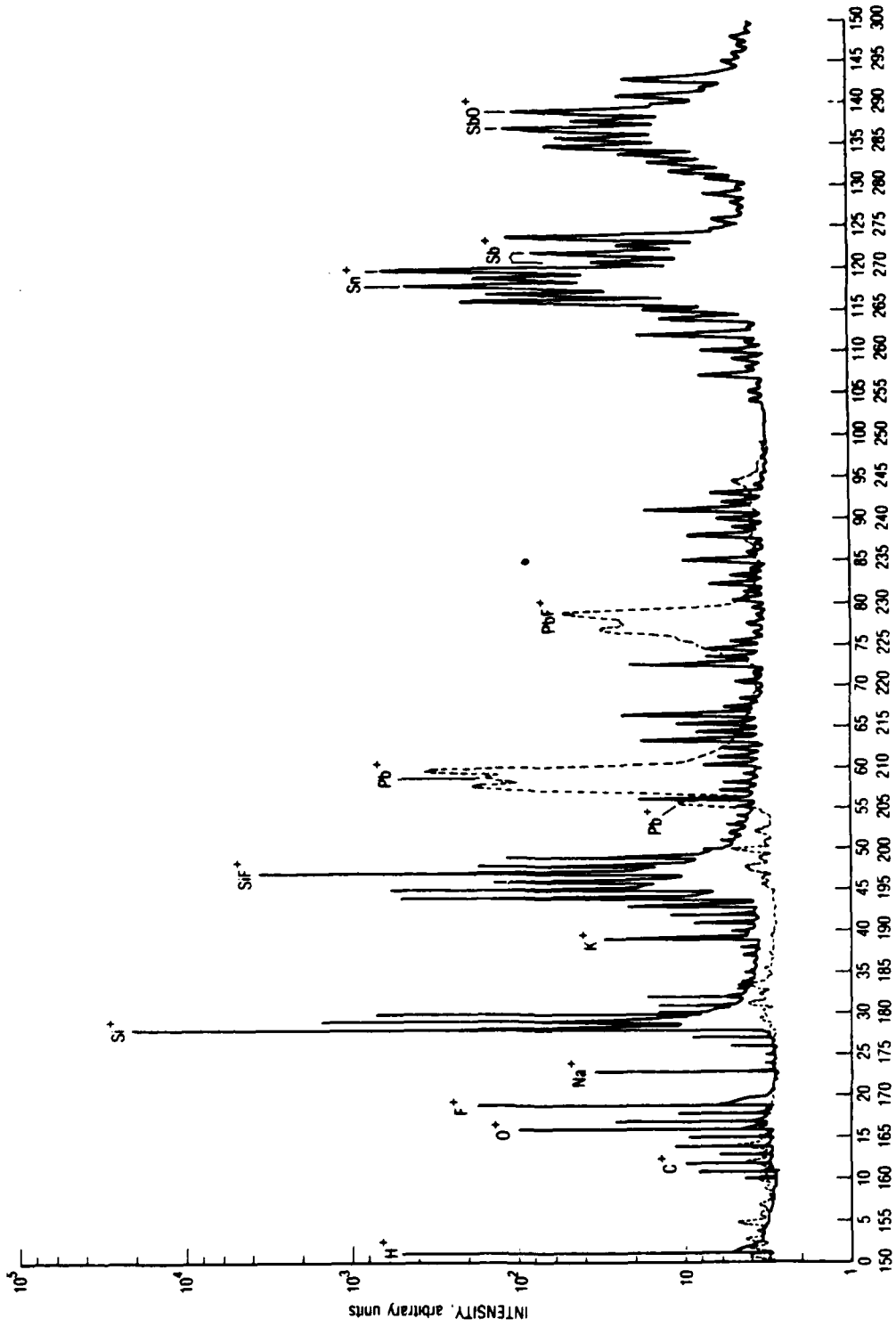


Figure 3. Secondary Ion Mass Spectra of Second-Stage Regenerator Balls, Unit S3

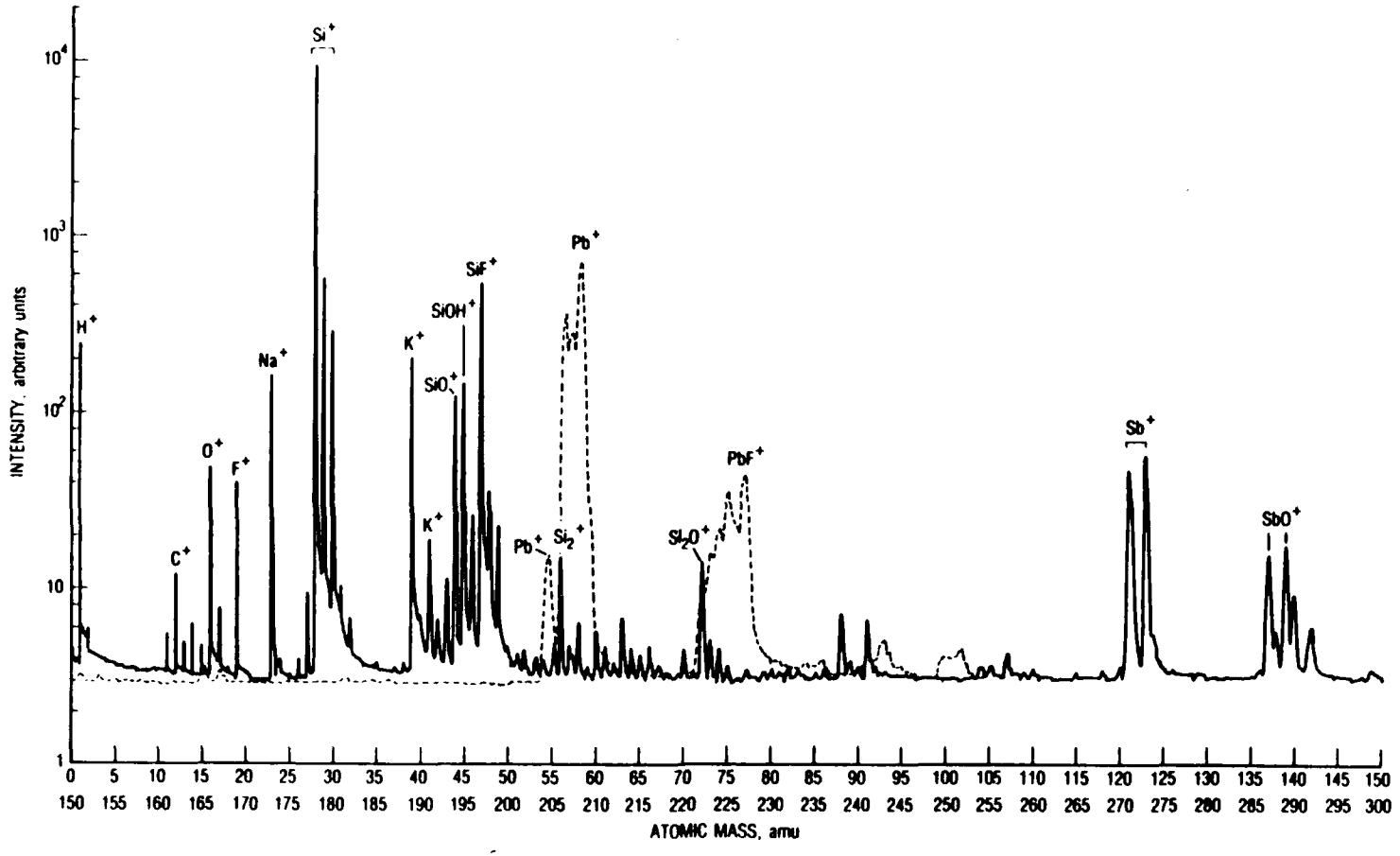


Figure 4. Secondary Ion Mass Spectra of Third-Stage Regenerator Balls, Unit S3

IV. DISCUSSION

Sample 7, taken from between the seals at the junction of the cold and hot cylinders, was identified as ZnO by XRD. The source of the Zn has not been clearly established, but it most likely originates from lubricating oils used in the system which often contain zinc stearate. Hughes personnel agreed that the oil might be the source of zinc.

Samples 48, 50, and 55 were taken from the first stage of the cold end and were identified as Mo-Nb primarily, and Fe-Cr-Ni, by SEM/EDXS analysis and as $Fe_2F_5 \cdot 7H_2O$ by XRD. Both results are consistent with degradation of the Inconel 718 used in the first-stage liner (see Table 2) and are similar to the materials found in the first stage of cryocooler unit S2 (Ref. 1). Sample 49, taken from the second stage, appears to consist of a crystalline phase of $Fe_2F_5 \cdot 7H_2O$ by XRD and Fe-Cr-Ni-containing material by EDXS, consistent with degradation of the 321 stainless steel used as the second- and third-stage liner material (Table 2). The EDXS results also show the presence of some silicates in all four of these samples. The silicates are part of the Fluorogold seal material, which is a glass-fiber-filled Teflon.

The metal fluorides appearing in these samples are probably the result of a chemical reaction between the Teflon of the seals and the metal liners. The XRD patterns are less complex for the S3 samples than for S2, indicating either a lesser amount of fluoride or a less active wear process. Either option can be attributed to the difference in seal pressures between the two units.

Samples 23, 58, 59, 62, 65, 73, and 75a, were taken from various portions of the hot ends. All results are consistent with degradation of the Inconel 718 liner material. The apparent amorphous nature of samples 23, 59, 65, 73, and 75a, and the identification of samples 59 and 65 as primarily Mo-Nb, indicates that a preferential removal of the Mo and Nb from the Inconel is occurring. It is not clear at present whether this preferential removal results from etching by a fluoride or from the wear process.

Table 2. Nominal Composition of Metal Alloys Used in Wearing Parts of Unit S3

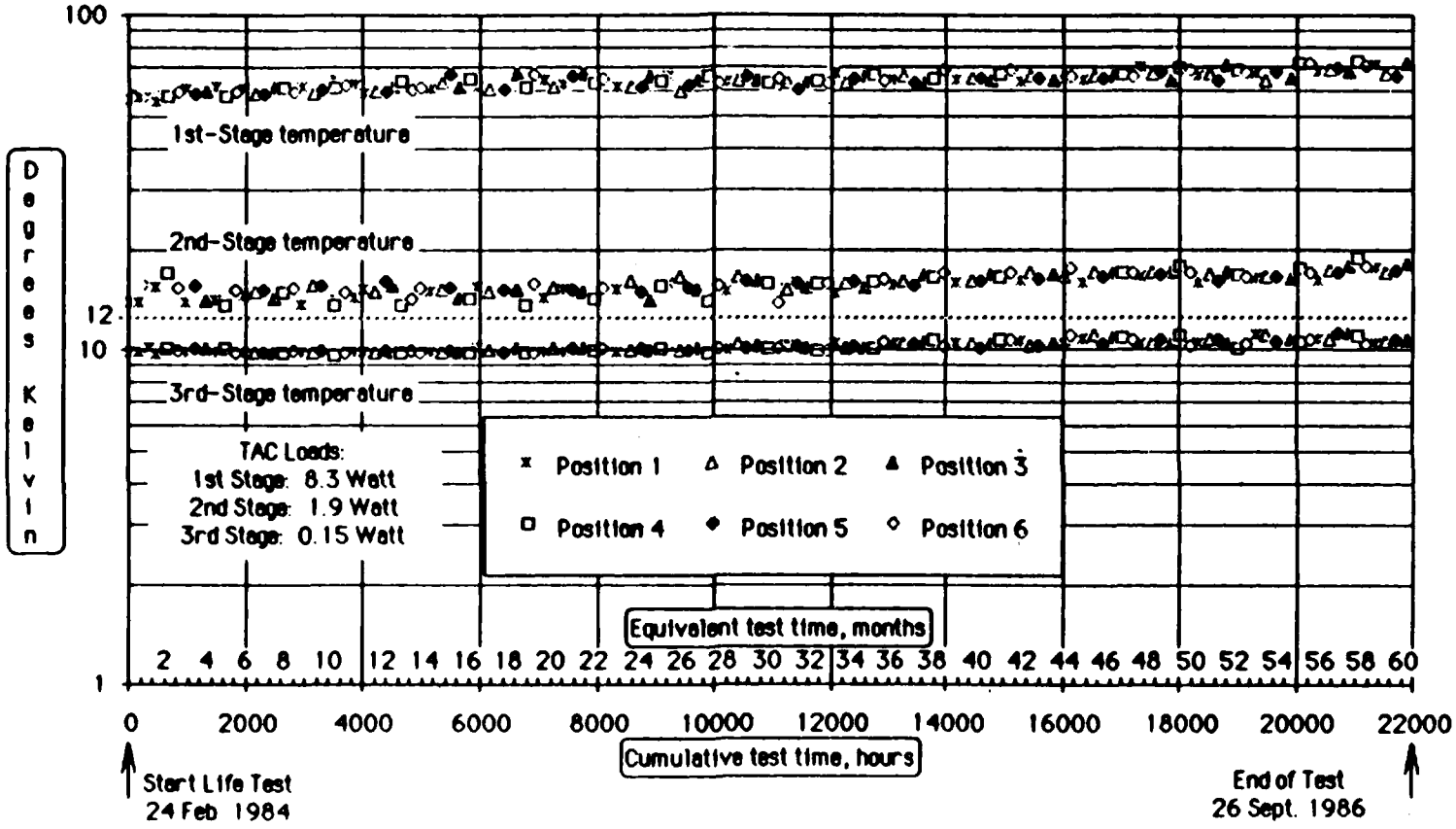
Inconel 718, wt%	Element	321 Stainless Steel, wt%
54.6	Ni	10
19	Cr	18
17	Fe	72
5	Nb	--
3	Mo	--
0.8	Ti	tr
0.6	Al	--

NOTE: Inconel 718 is used for the bore of the first-stage cold end and hot cylinder liner bore. 321 stainless steel is used for the second- and third-stage cold end bores.

The second-stage regenerator balls were 100 μm in diameter and were coated with Si and F, as was unit S2. The balls are made from Pb, Sb, and Sn, all of which appeared in the IMMA scan. The regenerator balls from the third stage were larger (250 μm diameter) and again showed large amounts of Si and F, along with Pb and Sb. No Sn was seen in the third-stage balls. The third-stage results are the same as those for unit S2.

Like unit S2, the cold stages contain Fluorogold, which is the most likely source of both the Si and F identified on the balls. Pb, Sb, and Sn are the components of the pristine balls.

The wear/degradation process which has occurred in unit S3 appears to be the same as that which occurred in unit S2, but the lower seal pressure in S3 has led to a slower rate of degradation. In both cryocoolers, the Delrin AF inlet rider material was severely degraded and is a likely source of CO/CO_2 gas, which will degrade the performance of the machines. Examination of the S3 Accelerated Life Test Performance History (Fig. 5) shows some degradation of the temperatures of each of the three stages. The first-stage steady-state temperature is about 10 K higher at the end of the test than it was at the beginning, whereas the second-stage temperature rose about 5 K and the third-stage rose about 0.5 K. These temperature rises may be the result of several factors, including changes in clearances resulting from wear of the seals and riders, and contamination of the working fluid as a result of the degradation of the Fluorogold and Delrin. The degree of possible contamination will be affected by the speed of the machine, the seal pressure, and the materials in the unit which can be attacked by the SiF_4. Those materials include, but are not limited to, the Delrin inlet rider, the glass fibers in the Fluorogold, and the metal liners and bores. It is not clear from the tear-downs of the two units whether the Delrin acts as a preferential scavenger of the fluoride (thus preventing degradation of more critical parts) or is only the most attacked material in the machine. Further laboratory investigation is required to determine which is the case.



SOURCE: Hughes Aircraft Company, El Segundo, CA.

Figure 5. S3 Accelerated Life Test Performance History (TAC Loads/300 rpm)

V. SUMMARY AND CONCLUSIONS

Debris samples from the Hughes VM cryocooler unit S3 have been analyzed. The debris consists of silicates, metals, and metal fluorides. These species originate from the glass in the seals and riders, liner bore and bore materials, and reacted metals from the bore areas. The results are essentially the same as those obtained previously from unit S2. A group of amorphous Mo-Nb metal particulates, however, is present in unit S3 that was not found in the accelerated contamination unit. The exact reason for the appearance of this material is not known but could result from either preferential etching by a fluoride species or from the natural wear process expected as the seals, riders, and liner materials interact.

Based on the results obtained from analysis of debris from both units, operated under different conditions, it appears that the five-year VM cryocooler will perform at its rated speed and power for the required lifetime. It has been demonstrated, however, that under more severe operating conditions, undesirable features exist which make the machine unforgiving in practice. In both units, the Fluorogold seals degraded in such a fashion as to introduce an extremely corrosive compound (SiF_4) into the working fluid. This corrosive agent attacks the materials in all parts of the machine and may adversely affect the working fluid properties. The presence of Delrin in the machine appears to be detrimental when used in conjunction with Fluorogold, because Delrin is severely attacked by the fluoride reaction product and forms CO/CO_2 during reaction with the fluoride. The CO/CO_2 is certain to have an adverse effect on the performance of the machine.

In order to upgrade the performance of this VM cryocooler design, certain changes should be made in the materials and additional tests using the new materials need to be conducted. The major materials changes involve substitution of different materials for the Delrin and Fluorogold, both of which contribute to degradation of performance, although the degree of synergism between Delrin and Fluorogold is not known. Additional required testing includes both the accelerated contamination and accelerated life tests. The

results of unit S2 are ambiguous regarding the effects of particulate contamination: The degradation mechanism exhibited by the machine was significantly different from what the test was designed to show. At least some of the particulates generated in this test reacted to form gaseous species rather than particulates capable of clogging the mechanical parts. With different materials, particulates may have a greater role in affecting the mechanical performance of the machine. In addition, lifetimes of the new seal and rider materials must be demonstrated in service-type conditions.

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